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1.0 INTRODUCTION

1.1 PURPOSE

The purpose of this integrated test plan is to guide the demonstration of the steam reforming technology being conducted in association with the Volatile Organic Compound-Arid Integrated Demonstration (VOC-Arid ID). This plan presents: (1) a background of the VOC-Arid ID and the 200 West Area Carbon Tetrachloride Expedited Response Action (ERA); (2) a description of the steam reforming technology; (3) objectives of the demonstration; and (4) a description of the demonstration tasks.

1.2 BACKGROUND

1.2.1 Programmatic

The demonstration described in this plan is being conducted as a coordinated effort between the ERA and the VOC-Arid ID. Steam reforming will be evaluated for the destruction of carbon tetrachloride (CCl_4) after desorption from granular-activated carbon (GAC) canisters (drums) as part of the VOC-Arid ID. The drums will be loaded with the CCl_4 from the ERA operation. Steam reforming is also being evaluated for the ERA as an alternate method for destroying CCl_4 retrieved from operations. Currently, the CCl_4 is loaded onto the GAC and the loaded GAC sent offsite for CCl_4 destruction and GAC reactivation. This is the present baseline method of treatment by which alternative methods will be compared. It is thought that the CCl_4 could be directly destroyed as it is extracted from the ground, or that carbon could be used as an intermediate step, enabling campaigning of the canisters through a destruction system. The objective of this type of evaluation would be to implement a less costly method treatment/destruction method for CCl_4 at the Hanford Site.

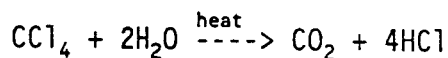
1.2.1.1 VOC-Arid ID. The VOC-Arid ID is one of several DOE integrated demonstrations designed to support the testing of emerging environmental management and restoration technologies. The principal objective of the VOC-Arid ID at the Hanford Site is to determine the viability of emerging technologies that can be used to characterize, remediate, and/or monitor arid or semiarid sites containing VOC (e.g., CCl_4) with or without associated metal and radionuclide contamination.

1.2.1.2 200 West Area Carbon Tetrachloride ERA. This ERA is being conducted by the U.S. Department of Energy (DOE) at the direction of the U.S. Environmental Protection Agency (EPA) and the Washington Department of Ecology (Ecology). An ERA is a removal action under the Comprehensive Environmental Response, Compensation, and Liability Act of 1980 (CERCLA), which allows ERA to be taken where early remediation will abate imminent hazards or prevent increased degradation that might occur if action were delayed until completion of a remedial investigation/feasibility study (RI/FS) and record of decision.

The 200 West Area CCl_4 ERA is based on concerns that the CCl_4 residing in the soils underlying the 200 West Area serves as a source of contamination to the groundwater. Thus, the purpose of the ERA is to minimize contaminant migration within the unsaturated soils in the 200 West Area by removing the CCl_4 in the vapor phase.

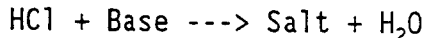
1.2.2 Technology

Steam reforming (Figure 1) destroys organics by reaction with superheated steam. When CCl_4 is mixed with steam and heated to 500°C or higher, CCl_4 decomposes to carbon dioxide (CO_2) and hydrochloric acid (HCl) as follows:



At higher temperatures, (800°C with a catalyst, 1200°C without), quantitative destruction (99.99%) has been achieved (Nimlos 1992, Galloway 1989a,b,c).

A moving bed evaporator is an integral part of the steam-reforming system. The moving bed is used to neutralize the HCl produced by the decomposition of CCl_4 . The neutralization is accomplished by coating the ceramic spheres of the moving bed with alkali base. The base then consumes the HCl released by the decomposition of CCl_4 by the following reaction:

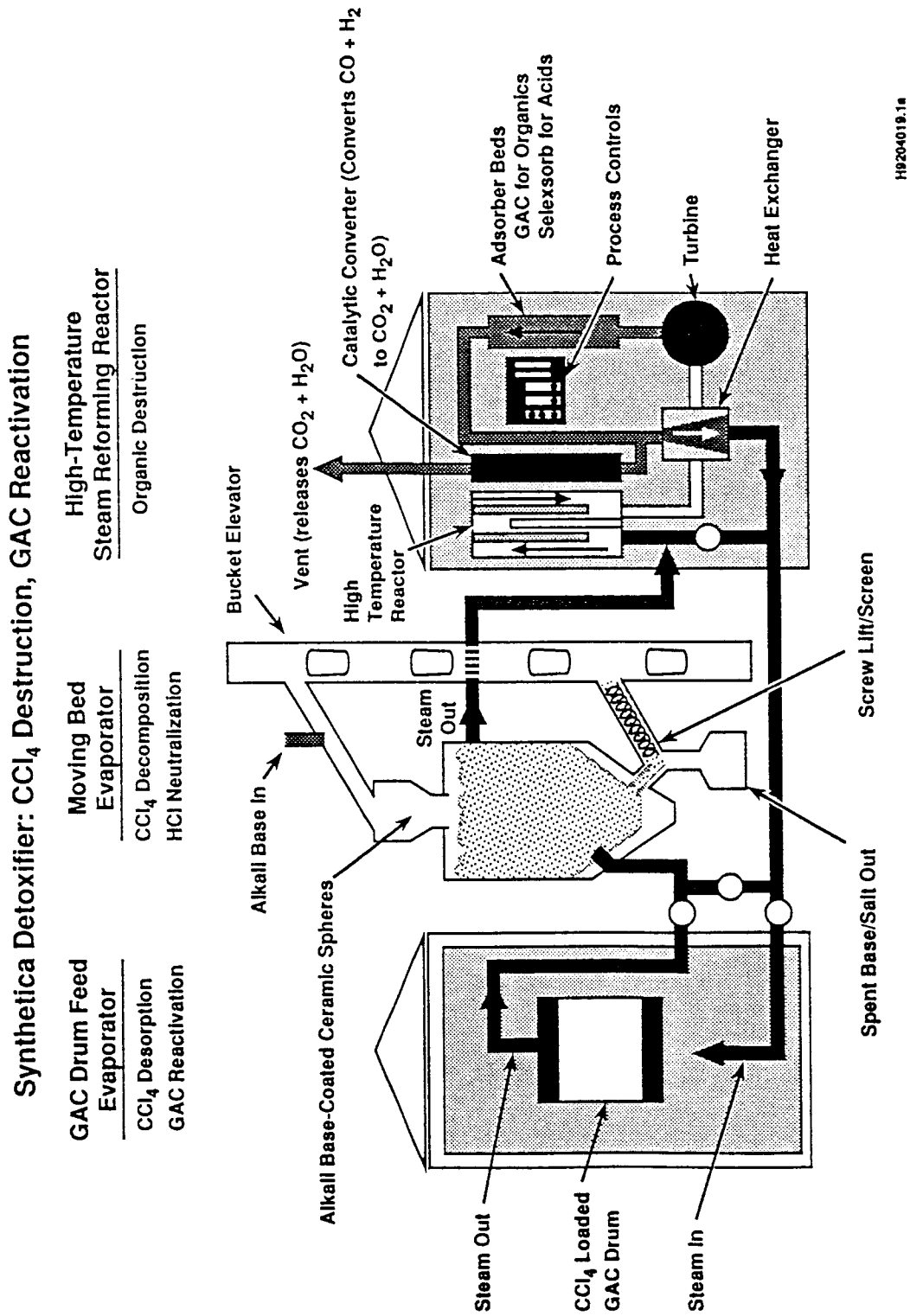


Because steam reforming is not a combustion process, oxides of nitrogen and products of incomplete combustion are not formed when organics are destroyed. Since heating of the CCl_4 in the absence of water/steam produces phosgene, control of the process is paramount. Equilibrium calculations predict complete conversion of phosgene to CO_2 and HCl at standard operating conditions. Because the reactor will not operate in the absence of steam, phosgene production during steam reforming should not be a problem.

Steam reforming of CCl_4 is a four step process. First, the adsorbed CCl_4 is desorbed from the GAC. Next, the CCl_4 is decomposed, releasing HCl , which is then neutralized. Then, any organic byproducts produced by the decomposition are destroyed by exposure to high temperatures. Finally, the GAC is reactivated. In theory, this would allow a closed-loop system wherein a vapor extraction system would collect vapors from a contaminated site, storing them in the GAC drum, then removing and destroying the organics while reactivating the carbon for reuse at the vapor extraction site.

A steam-reforming test is being conducted by Synthetica Technologies, Inc., Aiken, South Carolina, for the VOC Non-Arid ID (Savannah River) to test its capacity for the destruction of trichloroethylene (TCE). Because it is not obvious that steam reforming will also destroy a fully halogenated organic like CCl_4 with the same efficiency that it destroys a partially halogenated organic like TCE, this test on CCl_4 destruction will supplement the test being conducted for Savannah River.

Figure 1. Steam Reforming Process.



2.0 TEST OBJECTIVES

The objective of this demonstration is to obtain information on the removal of halogenated organics from GAC and the destruction of halogenated organics by steam reforming. The information will be used to compare this technology against a baseline. The principal investigator will present the results of this demonstration per the protocols set down by this test plan.

The specific test objectives are: (1) demonstrate 99.99% destruction of CCl_4 and chloroform; (2) demonstrate the absence of hazardous material air emissions (complete combustion/oxidation); (3) demonstrate the absence of CCl_4 in the spent base; and (4) demonstrate degree of reactivation of the activated carbon.

3.0 TEST DESCRIPTION

To demonstrate the objectives specified in Chapter 2, drums of GAC will be loaded with CCl_4 stripped from arid soils at Hanford and shipped to Synthetica Technologies, Inc., where existing and experimental technology will be used to desorb the CCl_4 from the GAC and to destroy the halogenated organics. It should be noted that other organics, such as chloroform, may be associated with the CCl_4 and will also require monitoring.

4.0 WORK TO BE CONDUCTED AT THE HANFORD SITE

4.1 SAMPLING OF CANISTERS PRIOR TO LOADING

Prior to loading the GAC with CCl_4 , samples will be taken to determine the adsorptive capacity of the carbon. All samples will be collected per the protocol set forth in the *Carbon Tetrachloride Vapor Extraction Project Radon Assessment Test Plan* (WHC 1992, Section 3.2). One composite sample will be taken from each drum. The samples will be shipped to Synthetica Technologies, Inc. and will be analyzed for adsorptive capacity.

4.2 ADSORPTION OF $\text{CCl}_4/\text{CHCl}_3$ ON GAC

Four 55-gal steel drums, each containing 180 lb of GAC, have been shipped by Synthetica Technologies, Inc. to Hanford to perform this demonstration. The ERA vapor extraction system, modified as necessary to fill 55-gal drums, will be used to load the GAC drums with CCl_4 extracted from the 200 West Area of the Hanford Site. Drums will be fitted with pressure gages to monitor the pressure after loading and during shipment.

5.0 WORK TO BE CONDUCTED AT SYNTHETICA

5.1 DESTRUCTION OF CHLOROCARBONS

Following the sorption of organics onto the GAC at Hanford, the drums will be shipped to Synthetica for processing. The removal/destruction of CCl_4 occurs in three stages. These stages are depicted in Figure 1 and further described in this section.

The chlorocarbons will be desorbed from the GAC by placing each drum in the drum feeder of the Synthetica Technologies, Inc. detoxifier and passing 260°C steam through the drum. The desorption of CCl_4 by the steam will be monitored by an experimental thin film sensor.

The chlorocarbon-laden steam (exhaust) produced by this desorption step will be passed at 700°C through a moving bed evaporator filled with ceramic spheres, for the initial CCl_4 decomposition. H_2 , CO , and HCl(g) will be generated, the latter being neutralized by reaction with alkali base, which is continuously injected into the evaporator. The CCl_4 and HCl content in the evaporator's effluent will be continuously monitored. Since the upper limit for chlorine in the detoxifier's feed stream is 2% chlorine by weight, if necessary, sufficient steam will be added to the effluent to reduce its chlorine levels below this amount.

The evaporator effluent will be piped to the detoxifier where the final stage of organic destruction is conducted at 1200°C . All remaining HCl will be removed. The CO and trace organic residues in the detoxifier effluent will be destroyed by passage through a catalytic converter.

5.2 SAMPLING AND ANALYSIS TASKS/ANALYSIS OF DETOXIFIER'S EFFLUENTS

Samples of the effluent, spent base/salts, and the GAC will be taken to determine the effectiveness of steam reforming. For this demonstration, the main objective is to determine if the detoxifier is effective in CCl_4 destruction. The type of samples to be taken to derive conclusions is described in Table 1.

Table 1. Samples Taken to Derive Conclusions.

| Sample | Species | Method |
|---|--|--|
| Detoxifier effluents (prior to entering catalytic converter) ^a | Volatile organics | Adsorption on charcoal NIOSH trap, GC/MS, EPA 8240 ^b |
| | Semivolatile organics | Adsorption on trap, GC/MS, EPA 8260 ^b |
| Emissions following catalytic converter | Fixed gasses (CO, CO ₂ , CH ₄ , O ₂ , N ₂) | Bag, GC/FID |
| | Halocarbons | Bag, GC/Hall |
| Spent salt (slurry base) | Nitrate | Ion chromatography, EPA 300.3 ^b |
| | Metals | TCLP, atomic adsorption |
| | Volatile organics | GC/MS, EPA 8240 ^b |
| | Semivolatile organics | GC/MS, EPA 8260 ^b |
| | Organic carbon | TOC, EPA 9060 ^b |
| | pH | |
| | Flash point | |
| Solid residues | Organic carbon | TOC, EPA 9060 ^b |
| GAC | Apparent density | ASTM |
| | Iodine # | ASTM |
| | Volatile material | ASTM |
| | Ash | ASTM |

^aEffluent will also be analyzed for phosgene and dichloroacetylchloride.

^bFrom Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846, U.S. Environmental Protection Agency, Washington, D.C.

ASTM = American Society for Testing and Materials.

FID = flame ionization detector

GC = gas chromatograph

MS = mass spectrometer

NIOSH = National Institute of Occupational Safety and Health

TCLP = Toxic Characteristic Leach Procedure

TOC = total organic carbon.

5.3 POST-TEST DATA ANALYSIS/INTERIM REPORT

After all four drums have been processed and all samples analyzed, the test procedures, analytical methods and results will be documented, and the technical performance of the detoxifier will be assessed. The principal investigator will provide a performance report addressing the demonstration of the objectives specified for this test.

- demonstrate 99.99% destruction of CCl_4 and chloroform
 - estimate cost/volume destroyed
 - completeness of destruction (effectiveness - absence of incomplete combustion products)
- demonstrate the absence of hazardous material air emissions - analytical results from the effluent sampling (before and after the stream flows through the catalytic convertor)
- demonstrate the absence of CCl_4 in the spent base
 - total volume of secondary waste produced (hazardous and non-hazardous)
 - type of waste generated (HCl , salts, etc.)
- demonstrate complete reactivation of the activated carbon - carbon adsorptive capacity following reactivation compared to adsorptivity prior to use of canisters.

The performance report shall include the following information:

- Technology Description
- Test Objectives
- Test Boundaries
- Test Parameters (Data Quality Objectives)
- Materials and Methods
- Experiment Results (did demonstration meet objectives?)
- Conclusions and Lessons learned
- Recommendations for follow-up work.

6.0 DATA MANAGEMENT AND REPORTING

6.1 FOR WORK CONDUCTED AT HANFORD

A data management plan has been prepared for data collection and analysis associated with the ERA data objectives (Rohay 1991) and VOC-Arid ID data objectives (Last and Rohay 1991). Data collected by the Demonstration Operation in support of the technology demonstration and testing will comply with Environmental Investigations Instruction (EII) 1.6, Records Management (WHC 1988a).

6.2 FOR WORK CONDUCTED AT SYNTHETICA

A letter report describing the lessons learned during the test of the system will be provided to the Demonstration Operations following the test. The analysis data will also be summarized and evaluated as directed in Section 5.3.

7.0 QUALITY ASSURANCE

The objective of this integrated test plan is to ensure that the data obtained and the conclusions drawn are sufficiently accurate and reliable to support decisions associated with the evaluation of the demonstration.

To ensure that the field demonstration activities conducted at Hanford are consistent with DOE-RL Order 5700.6C, *Quality Assurance*, all work will be performed in compliance with Westinghouse Hanford's QA manual (WHC 1988b) and with applicable procedures outlined in the QA program Plan (WHC 1990); this QA program plan describes the various plans, procedures, and instructions that will be used by Westinghouse Hanford.

The sampling performed at Synthetica Technologies, Inc. will be conducted according to EPA protocol, and the samples will be sent for analysis at an EPA certified laboratory.

8.0 REGULATORY AND HANFORD COMPLIANCE

8.1 TRANSPORTATION/SHIPPING OF MATERIALS OFFSITE

The GAC drums will be shipped off-site as treatability study samples. All offsite shipments will follow the requirements of 40 CFR 261.4(e) and Washington Administrative Code (WAC) 173-303-071(r). A review of the results from the radon assessment test will be required to determine if the GAC will require onsite disposal.

8.2 NATIONAL ENVIRONMENTAL POLICY ACT

The National Environmental Policy Act (NEPA), 42 USC 4321-4370a is the basic federal charter for protecting the nation's environment. NEPA's focus is to ensure that federal agencies such as DOE give appropriate consideration to environmental impacts in their decision making. DOE has prepared an Engineering Evaluation/Cost Analysis - Environmental Assessment (EE/CA-EA) (DOE-RL 1991) which examined and compared the environmental impacts of the ERA's proposed removal and treatment activities, including use of offsite steam reforming as a means of vapor treatment. Based on the findings of that EE/CA-EA, DOE issued a Finding of No Significant Impact (FONSI) that determined that the preparation of an environmental impact statement for those activities is not required. Because both the activities described in this test plan and the potential environmental impacts associated with those

activities are identical in kind and scope to those activities described in the ERA EE/CA-EA and the FONSI, no further NEPA review or documentation is required.

8.3 SAFETY

Activities conducted during the collection of CCl_4 , the sampling of the canisters, and the on-site demonstration will be governed by site safety plans and other documents WHC. Health physics technicians and site safety officers will monitor for contamination as directed by the approved plans. A hazards classification of the activities covered in this test plan determined that they are in a general-use category (Lehrschall 1992). Access to the work site will be controlled by the field team leader. Training requirements are outlined in the *Hazardous Waste Operation Plan for the 200 West Area Carbon Tetrachloride Expedited Response Action* (Tuttle 1992).

The safety of work conducted at Synthetica is controlled solely by Synthetica Technologies, Inc.

8.4 WASTE DISPOSITION

Subtitle C of the Resource Conservation and Recovery Act of 1976 (RCRA), 42 USC 6921-6939b, establishes a comprehensive program to regulate newly generated hazardous waste. Administered by Ecology and EPA, RCRA Subtitle C requirements are contained in WAC 173-303 and in 40 CFR Parts 260 through 272 and apply to the generation, accumulation, treatment, storage, and disposal of hazardous waste. Hazardous waste generated at the Hanford Site as a result of the activities described herein will be handled according to EII 4.2, Interim Control of Unknown, Suspected Hazardous, and Mixed Waste (WHC 1988a).

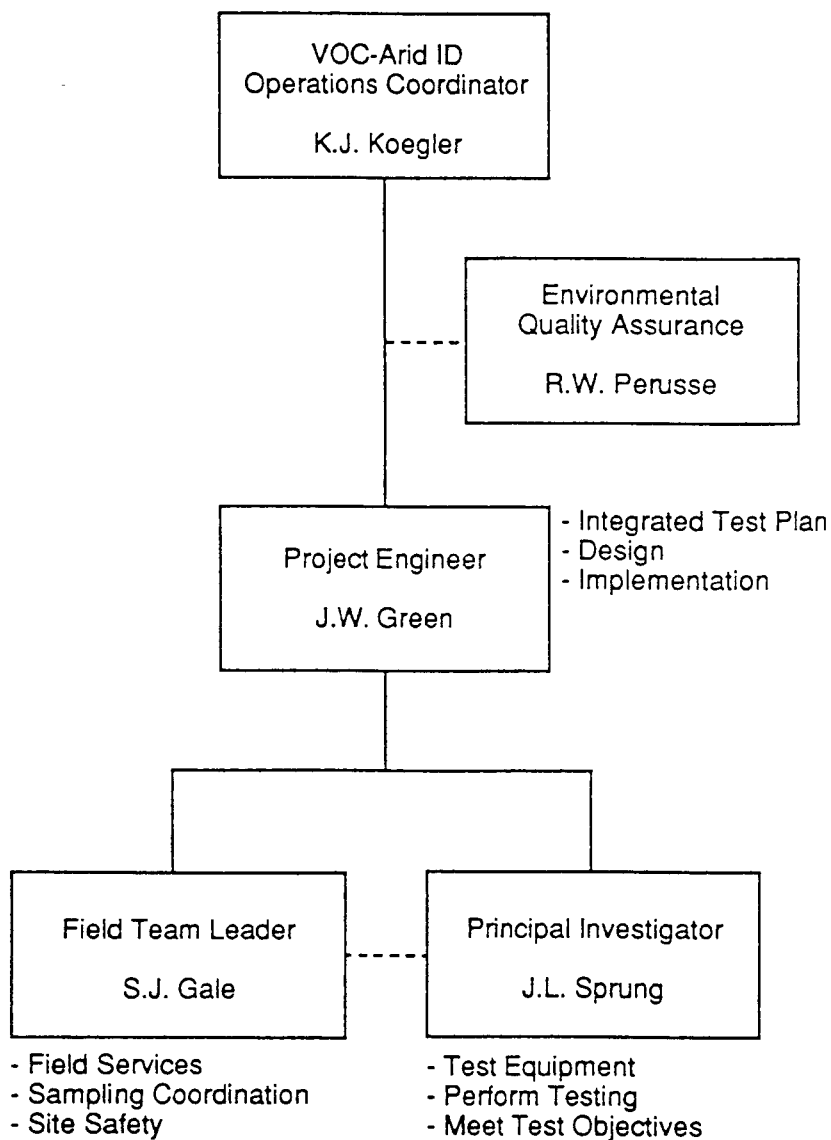
8.5 SYNTHETICA'S REGULATORY COMPLIANCE

The development of the Synthetica etoxifier was partially funded by the California Hazardous Waste Reduction Grant Program (Contract #86-T0119 A-1). Detoxifier effluents meet all California and EPA emissions standards. California and EPA staff inspected its performance during a 1989 site visit. Based on measured emissions and the design and performance of detoxifier safety systems, the California EPA approved the use of the detoxifier in on-site demonstrations. The waste destruction test specified in this document will be conducted in the laboratories of Synthetica Technologies, Inc. Synthetica has an exemption from the EPA, which allows limited quantities (< 500 lb) of waste materials to be used in onsite experiments to demonstrate the capabilities of the detoxifier. For such experiments, no additional approvals or permits is required. In addition, Synthetica has obtained a California New Technology Grant Permit, a Bay Area Air Quality Management District Permit, and EPA RD&D, California RD&D, EPA RCRA Part B Amendment, California TSDF Amendment, and California TTU permits are all in advanced stages of the approval process.

9.0 ORGANIZATION

Activities conducted under this integrated test plan will be conducted by Demonstration Operations in coordination with the appropriate principal investigator. Demonstration Operations is responsible for site characterization, engineering and conduct of field demonstrations, and regulatory and DOE/Hanford compliance. Reporting of the technology test evaluation will be the responsibility of the principal investigator. An organization chart is provided in Figure 2.


Figure 2. Field Demonstration Organization.



JMF/M062692-F

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| | | | WHC- 5D -EN-TP-010, Rev. 0 | | |
| | | | List attachments. none | | |
| | | | Date Release Required 8/28/92 | | |
| Title Test Plan for the Destructruction of Halogenated Organics by Steam Reformation | | | | Unclassified Category UC- | |
| | | | | Impact Level 3S Q | |
| New or novel (patentable) subject matter? <input checked="" type="checkbox"/> No <input type="checkbox"/> Yes If "Yes", has disclosure been submitted by WHC or other company? <input type="checkbox"/> No <input type="checkbox"/> Yes Disclosure No(s). | | | Information received from others in confidence, such as proprietary data, trade secrets, and/or inventions? <input checked="" type="checkbox"/> No <input type="checkbox"/> Yes (Identify) | | |
| Copyrights? <input checked="" type="checkbox"/> No <input type="checkbox"/> Yes If "Yes", has written permission been granted? <input type="checkbox"/> No <input type="checkbox"/> Yes (Attach Permission) | | | Trademarks? <input checked="" type="checkbox"/> No <input type="checkbox"/> Yes (Identify) | | |
| Complete for Speech or Presentation | | | | | |
| Title of Conference or Meeting | | | Group or Society Sponsoring | | |
| Date(s) of Conference or Meeting | | City/State | | Will proceedings be published? <input type="checkbox"/> Yes <input type="checkbox"/> No Will material be handed out? <input type="checkbox"/> Yes <input type="checkbox"/> No | |
| Title of Journal | | | | | |
| CHECKLIST FOR SIGNATORIES | | | | | |
| Review Required per WHC-CM-3-4 | | Yes No | | Reviewer - Signature Indicates Approval | |
| | | | | Name (printed) Signature Date | |
| Classification/Unclassified Controlled Nuclear Information | | <input type="checkbox"/> <input checked="" type="checkbox"/> | | <u>S. W. BERGLIN</u> <u>[Signature]</u> <u>10/6/92</u> | |
| Patent - General Counsel | | <input checked="" type="checkbox"/> <input type="checkbox"/> | | <u>B. Williamson</u> <u>[Signature]</u> <u>10/6/92</u> | |
| Legal - General Counsel | | <input checked="" type="checkbox"/> <input type="checkbox"/> | | | |
| Applied Technology/Export Controlled Information or International Program | | <input type="checkbox"/> <input checked="" type="checkbox"/> | | | |
| WHC Program/Project | | <input type="checkbox"/> <input checked="" type="checkbox"/> | | | |
| Communications | | <input type="checkbox"/> <input checked="" type="checkbox"/> | | | |
| RL Program/Project | | <input type="checkbox"/> <input checked="" type="checkbox"/> | | | |
| Publication Services | | <input checked="" type="checkbox"/> <input type="checkbox"/> | | <u>L. Hermann</u> <u>[Signature]</u> <u>10/9/92</u> | |
| Other Program/Project | | <input type="checkbox"/> <input checked="" type="checkbox"/> | | | |
| Information conforms to all applicable requirements. The above information is certified to be correct. | | | | | |
| References Available to Intended Audience | | Yes No | | INFORMATION RELEASE ADMINISTRATION APPROVAL STAMP Stamp is required before release. Release is contingent upon resolution of mandatory comments.  | |
| Transmit to DOE-HQ/Office of Scientific and Technical Information | | <input type="checkbox"/> <input checked="" type="checkbox"/> | | | |
| Author/Requestor (Printed/Signature) | | Date | | | |
| J.M. Frain <u>[Signature]</u> | | 8/20/92 | | | |
| Intended Audience | | | | Date Cancelled _____ Date Disapproved _____ | |
| <input type="checkbox"/> Internal <input type="checkbox"/> Sponsor <input checked="" type="checkbox"/> External | | | | | |
| Responsible Manager (Printed/Signature) | | Date | | | |
| M.C. Hagood <u>[Signature]</u> | | 8/20/92 | | | |